

इंटरनेट

मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

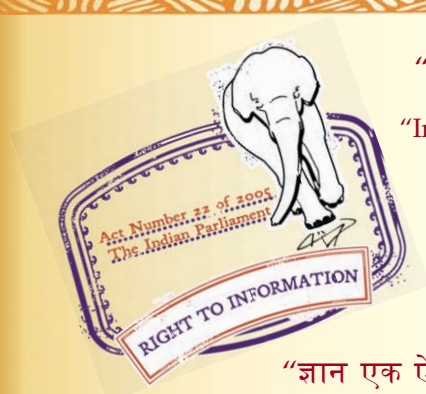
“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 1917-6 (1992): Chemical analysis of quartzite and high silica sand, Part 6: Determination of calcium and magnesium by atomic absorption spectrometric method [MTD 13: Ores and Raw Materials]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE



भारतीय मानक

उच्चसिलिका बालू एवं क्वार्ट्ज़ाइट का
रासायनिक विश्लेषण

भाग 6 परमाण्वीय अवशोषण पद्धति द्वारा कैल्शियम और
मैग्नीशियम ज्ञात करना

(पहला पुनरीक्षण)

Indian Standard

CHEMICAL ANALYSIS OF QUARTZITE
AND HIGH SILICA SAND

PART 6 DETERMINATION OF CALCIUM AND MAGNESIUM BY ATOMIC
ABSORPTION SPECTROMETRIC METHOD

(*First Revision*)

UDC 553.57 : 543.42 [546.41 + 546.46]

© BIS 1992

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

FOREWORD

This Indian Standard (Part 6) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ores, Minerals and Allied Materials for Metallurgical Industry Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 1917 : 1962 'Methods of Chemical Analysis of Quartzite and High Silica Sand' covers the methods for the determination of silica and other constituents of quartzite and high silica sand. The committee decided to revise this standard into different parts, and covering determination of each element in a separate part, which on publication would supersede the determination of that element given in IS 1917 : 1962. The determination of loss on ignition, silica, sodium, potassium, aluminium, iron, calcium, magnesium and titanium oxide in quartzite and high silica sand have been covered in this series.

This part covers the determination of calcium and magnesium. The other parts in this series are;

- Part 1 Determination of loss on ignition
- Part 2 Determination of sodium and potassium
- Part 3 Determination of silica
- Part 4 Determination of aluminium
- Part 5 Determination of iron
- Part 7 Determination of titania

In this revision the determination of calcium oxide by the 'Oxalate (volumetric) method' and magnesium by the 'Oxine Method' have been replaced by atomic absorption spectrometric method.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

CHEMICAL ANALYSIS OF QUARTZITE AND HIGH SILICA SAND

PART 6 DETERMINATION OF CALCIUM AND MAGNESIUM BY ATOMIC ABSORPTION SPECTROMETRIC METHOD

(*First Revision*)

1 SCOPE

1.1 This standard (Part 6) covers the method for determination of calcium (as CaO) and magnesium (as MgO) in quartzite and high silica sand in the range up to 0.5 percent by atomic absorption spectrometric method.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

<i>IS No.</i>	<i>Title</i>
1070 : 1977	Water for general laboratory use (<i>second revision</i>)
1811 : 1984	Methods of sampling foundry sands (<i>first revision</i>)
2245 : 1962	Methods of sampling quartzite

3 SAMPLING

3.1 The sample shall be drawn and prepared in accordance with IS 2245 : 1962 or IS 1811 : 1984 as the case may be.

3.2 Grind 3 to 5 g of the sample (**3.1**) so that it passes through IS test sieve No. 15 (145 micron). Dry the sample in a weighing bottle at 105° to 110°C for two hrs. Keep the bottle stoppered in a desiccator and cool to room temperature.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070 : 1977) shall be employed for test.

5 DETERMINATION OF CALCIUM AND MAGNESIUM

5.1 Outline of the Method

The sample is decomposed by hydrofluorization and the residue fused with potassium bisulphate

and dissolved in hydrochloric acid. Calcium and magnesium are determined at 422.7 and 285.2 nm respectively by atomic absorption spectrometric method using lanthanum chloride as releasing agent.

5.2 Reagents

5.2.1 *Hydrofluoric Acid*, 40 percent.

5.2.2 *Dilute Sulphuric Acid*, 1 : 1 (v/v).

5.2.3 *Potassium Bisulphate*, Solid.

5.2.4 *Dilute Hydrochloric Acid*, 1 : 1 (v/v).

5.2.5 *Stock Calcium Solution* (1 000 µg/ml).

Dissolve 2.497 g of calcium carbonate in 50 ml of dilute hydrochloric acid. Boil the solution to remove carbon dioxide, cool and make up to 1 000 ml in a volumetric flask with water.

5.2.5.1 *Standard calcium solution* (50 µg/ml)

Take 5 ml of stock calcium solution **5.2.5** and dilute to 100 ml. This solution contains 50 µg of calcium per ml.

5.2.6 *Stock Magnesium Solution* (1 000 µg/ml)

Dissolve 1.00 g of magnesium metal in 50 ml of dilute hydrochloric acid (1 : 1) and make up volume in 1 litre volumetric flask.

5.2.6.1 *Standard magnesium solution* (50 µg/ml)

Take 5 ml of stock magnesium solution (**5.2.6**) and dilute to 100 ml in volumetric flask. This solution contains 50 µg of magnesium per ml.

5.3 Apparatus

5.3.1 *Atomic Absorption Spectrometer*

Equipped with a monochromatic radiation sources such as calcium and magnesium hollow cathode lamps, a monochromator to isolate the 422.7 and 285.2 nm resonance lines, an atomization source such as a burner and a readout device.

5.3.2 Operating Parameters

5.3.2.1 Hollow cathode lamps, calcium and magnesium

5.3.2.2 Wavelength, 422.7 nm for calcium and 285.2 nm for magnesium.

5.3.2.3 Flame, Air — acetylene.

5.3.2.4 Band pass, As specified by the manufacturer.

NOTE — Other operating parameters to be followed according to manufacturers instructions.

5.4 Procedure

5.4.1 Test Portion

Weigh to the nearest 0.001 g, 2.0 g of the sample. Transfer it to a platinum crucible.

5.4.2 Dissolution of the Test Portion

5.4.2.1 Moisten with 2 ml of water and add 10 ml of hydrofluoric acid (40 percent) and 1 ml of dilute sulphuric acid (1 : 1). Warm on a hot plate with the lid till the sample is decomposed. Remove the lid and evaporate slowly to fumes. Cool the dish and wash the inner sides of the dish with little water, add further 5 ml of hydrofluoric acid and evaporate on hot plate to dryness. Fuse the residue with 1 g of potassium bisulphate slowly to a clear melt. Cool and extract the melt with water and add 10 to 15 ml of dilute hydrochloric acid (1 : 1). Boil the extracted mass to get a clear solution. Cool and make up to 250 ml in a calibrated volumetric flask.

5.4.2.2 For calcium — Take an appropriate aliquot of the solution (5.4.2.1) containing not more than 5 µg of calcium per ml, add 2 ml of lanthanum chloride (10 percent) and make up the volume to 100 ml in a volumetric flask.

5.4.2.3 For magnesium — Take an aliquot of the solution 5.4.2.1 containing not more than 0.5 µg magnesium per ml, add 2 ml of lanthanum chloride (10 percent) and make up the volume to 100 ml in a volumetric flask.

5.4.3 Preparation of Calibration and Blank Solutions

5.4.3.1 (a) For calcium — Take six numbers 100 ml volumetric flask and to each flask add 0, 2, 4, 6, 8 and 10 ml of standard solution (5.2.5.1). Add 2 ml of lanthanum chloride solution (10 percent). Dilute to the mark and mix well. The prepared solutions contain calcium

in the range from 0 to 5 µg per ml. The zero member of this series will serve as the reference solution.

5.4.3.1 (b) For magnesium — Take six numbers of 100 ml volumetric flasks and to each flask add 0, 0.2, 0.4, 0.6, 0.8 and 1.0 ml of standard solution (5.2.6.1), add 2 ml of lanthanum chloride (10 percent) to each and make up the volume to 100 ml in a volumetric flask. The prepared calibration solutions contain magnesium in the range from 0.1 to 0.5 µg/ml.

5.4.3.2 Prepare a blank solution using same quantities of all the reagents and following the procedure specified in 5.4.2 but without the sample.

5.4.4 Adjustment of the Atomic Absorption Spectrometer

Follow the instructions of the manufacturer in preparing the instrument. Switch on the instrument and the required hollow cathode lamp. Fit the correct burner for air-acetylene flame and light the appropriate flame. Wait for about 20 minutes for stabilization. Set the wavelength at 422.7 nm for calcium or at 285.2 nm for magnesium. Optimise instrument response separate for calcium and magnesium by adjusting the wavelength, fuel, air, burner and nebulizer while aspirating the highest calibration solution of the particular element. As the sensitivity varies from instrument to instrument, the concentration of the standard series and of the test solution should be adjusted accordingly. At the same time check the linearity of the calibration curve.

Aspirate water and one of the calibration solution repeatedly to ensure that there is no drift of absorbance. Finally aspirate water and set the absorbance to zero reading.

5.4.5 Atomic Absorption Measurement

5.4.5.1 Aspirate first the blank solution and then the calibration solution of the element to be estimated in increasing order, aspirating water between each aspiration of the solution and record the absorbance reading. Then aspirate the test sample and note absorbance. Each aspiration should be made at least three times and take the average value. Solids which build up on the burner slit must be removed, otherwise they will cause a decrease of sensitivity.

5.4.5.2 Deduct the blank value from the absorbance of the standard solution as well as from the test solution.

5.4.5.3 Prepare a calibration curve by plotting the corrected absorbance values of the standard

solutions against the concentration in $\mu\text{g/ml}$ of the element. where

5.4.5.4 Compute the concentration of the test solution corrected for the blank to μg of element per ml from the calibration curve.

5.4.6 Calculations

5.4.6.1 Calculate the percentage of calcium or magnesium content as follows:

$$\text{Element, percent by mass} = \frac{A \times 100}{B \times 10^6}$$

A = concentration in $\mu\text{g/ml}$, of element, and

B = mass in g, of the sample represented by the aliquot taken in the final test solution.

5.4.6.2 From the value of the calcium and magnesium content as obtained in **5.4.6.1** calculate the percentage of calcium oxide and magnesium oxide as follows:

$$\begin{aligned} \% \text{ CaO} &= \% \text{ Ca} \times 1.39 \\ \% \text{ MgO} &= \% \text{ Mg} \times 1.66 \end{aligned}$$

Standard Mark

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

Bureau of Indian Standard

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 1986* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Revision of Indian Standards

Indian Standards are reviewed periodically and revised, when necessary and amendments, if any, are issued from time to time. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition. Comments on this Indian Standard may be sent to BIS giving the following reference:

Doc : No MTD 29 (3845)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices :

	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	{ 311 01 31 331 13 75
Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola CALCUTTA 700054	37 86 62
Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036	53 38 43
Southern : C. I. T. Campus, IV Cross Road, MADRAS 600113	235 02 16
Western : Manakalaya, E9 MIDC, Marol, Andheri (East) BOMBAY 400093	6 32 92 95
Branches : AHMADABAD, BANGALORE, BHOPAL, BHUBANESHWAR, COIMBATORE, FARIDABAD, GHAZIABAD, GUWAHATI, HYDERABAD, JAIPUR, KANPUR, LUCKNOW, PATNA, THIRUVANANTHAPURAM.	